AUSTRALIAN JOURNAL OF BASIC AND APPLIED SCIENCES

ISSN:1991-8178 EISSN: 2309-8414
Journal home page: www.ajbasweb.com

Effect of Organic Zinc Coating Layers on Steel Corrosion Behaviour

[1]Engku Norfatima Engku Dahalan, [1]Azizul Helmi Sofian, [3]Arman Abdullah, [2]Norhazilan Md Noor, [1]Abdul Halim Hadi Che Mohammad Yahya

[1]*Faculty of Chemical Engineering & Natural Resources, Universiti Malaysia Pahang, 26300 Gambang, Pahang, Malaysia.*
[2]*Faculty of Civil Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia.*

Address For Correspondence:
Azizul Helmi Sofian. Universiti Malaysia Pahang, Faculty of Chemical Engineering & Natural Resources, 26300. Gambang. Malaysia. +609-5492866 and azizulh@ump.edu.my

ARTICLE INFO

Article history:
Received 19 August 2016
Accepted 10 December 2016
Published 31 December 2016

Keywords:
Organic zinc coating, Corrosion behaviour, Layers, Pigment volume concentration, Potentiostat,Corrosion rates.

ABSTRACT

Throughout proper protection, preservation of metals in their pure form can be achieved. One of the possible methods is protection by organic zinc coating. This study was to compare the corrosion behaviour with different layers of coating between formulated organic zinc coating and commercialized spray. The pigment volume concentration (PVC) was diversified to 10, 20 and 30% with different layers of coating. The corrosion behaviour of coated sample was tested by using potentiostat. The results of polarization measurement show that the linear polarization resistance was significantly influenced by the pigment content and the layers of coating. The lowest corrosion rates for formulated organic zinc coating and commercialized spray were 0.00018 and 0.0062 mm/yr respectively, both from 5 layers of coating. 5 layers of coating give a better protection towards corrosion in corrosive environment.

INTRODUCTION

The corrosion failure of engineering materials has brought a great deal of economy loss, so that more researchers have focused on the study of corrosion behaviour and mechanism of metal materials to find better protective methods and technologies (Rousseau *et al.*, 2009) and (Rios *et al.*, 2014). Organic coatings are most popular method and widely used in protection of metallic materials (Rosales *et al.*, 2004) which ensures both a chemical and electrochemical reaction between an anticorrosion pigment and the metal or a corrosion environment penetrating the coating (del Amo *et al.*, 2004). In order to provide electric conductivity between adjacent particles as well as between the pigment particles and the substrate, a high pigment concentration is necessarily needed, so that it will packed closely enough in the dry film. This allows the zinc-steel galvanic couple to generate the conduction of electric current during electrochemical reaction. As a result, a very porous film will be obtained. Therefore, it will be able to absorb the penetrating solution (Shreepathi *et al*., 2010).

In organic coatings, a crosslinking (curing) reactions forming a highly crosslinked polymeric film, and a binder also presents to coat the zinc particles. The resistivity of the coatings is increased to the point where its electrical conductivity decreased below the critical level even until the coating material does not provide electrochemical protection anymore (Buxbaum, 1998). Therefore, the electric conductivity of pigmented film is related to the PVC of the zinc in the coating material binder. Zinc particle is efficient and achieved the highest electric conductivity at concentration ranging from 94% to 96% with binder content at 5-8% in paints (Arianpouya *et al*., 2013). However, this requirement comes with several problems. Such low binder content is insufficient to attain the required physio-mechanical properties like adhesion, impact resistance and cupping

To Cite This Article: Engku Norfatima Engku Dahalan, Azizul Helmi Sofian, Arman Abdullah, Norhazilan Md Noor, Abdul Halim Hadi Che Mohammad Yahya., Effect of Organic Zinc Coating Layers on Steel Corrosion Behaviour. ***Aust. J. Basic & Appl. Sci.,*** *10(17): 40-47, 2016*

resistance. Physico-chemical properties and corrosion resistance of zinc-rich coating are depending on the PVC (Vilche *et al*., 2002).

Under immersion conditions, the time of cathodic protection depends on the zinc content in the coating film, and they verified coatings with 60% PVC of zinc powder showed good corrosion resistance mainly due to the cathodic protection. Later on, some researchers stated that the PVC of 65% and above (more than or equal to 90% by weight on dry film) will build a reliable percolation (Nanna and Bierwagen, 2004) and (Bierwagen *et al*., 2007). (Shreepathi *et al*., 2010) clearly stated based on EIS studies that percolation limit for providing sufficient galvanic protection was attained at 70% zinc on dry film. When the zinc content in the coatings excess 80%, superior galvanic protection is achieved. However, coating containing 40% of zinc content by weight also shows very good protection mechanism mainly from epoxy resin as a binder.

The organic binder and inorganic pigments/fillers in this coatings provide barrier and active protection to the metal substrate from corrosion. The binder and filler in the organic coatings contribute to the barrier protection, while active protection was achieved from anticorrosive pigments (Liu *et al*., 2016); (Visser *et al*., 2015). Zinc powder was known as good sacrificial protection long time ago. Zinc powder is among those significant non-toxic metal pigments (Barranco *et al*., 2004), which has barrier and electrochemical mechanism protection (Prokes and Kalendova, 2007).

Hence, the aim of this paper is to study the effect of coating layers on corrosion behaviour by formulating a low PVC to eliminates a problem regarding porosity and adhesion of coating. The properties of the coating can be controlled by controlling the composition of the pigments. This can be achieved by adjusting the volume percentage of zinc pigments. The low PVC with high content of binder will be expected to have good barrier protection, thus lowering the value of corrosion rates.

MATERIALS AND METHOD

Materials:

In this study, the substrates used for all coating substrates are the carbon steel sheet. The anticorrosion pigment used in this study is zinc oxide based on the its barrier and electrochemical mechanism protection. While kaolin (China clay) is used as an extender pigment because it can extend the duration of the cathodic protection of coating. Araldite epoxy resin was used to formulate anticorrosion paint. Epoxy resin plays a significant role in the formulation of coating. It provides excellent adhesion to metals, good chemical resistance and great mechanical properties. Ethanol was used as a solvent to dissolve all the pigments and epoxy resin. All chemicals were supplied from Sigma Aldrich.

Pretreatment of metal substrate:

The iron sheet was cut into (20mm X 20mm X 0.5mm) in dimension. The metallic substrates were first serially polished with sand papers #180, #220, #280, #600, #800, #1000 and #1500 to produce a clean and shiny surface. After that, the metal substrates were then introduced into ethanol and ultra-sonicated for 30 minutes. Ethanol is employed to degrease the surface of the metal substrates. The clean substrates then, dried at room temperature before proceed to the next stages.

Copper wire was cut to 10 cm in length and then soldered on the back of the substrate plate. Copper wire is being used because it can conduct electricity during a corrosion test. After that, epoxy resin was mounted on the soldered part to strengthen the adhesion of copper wire on the substrate plate. The substrates with mounted epoxy resin were dried at room temperature for 6 to 7 hours. The dried samples were stored in the oven at 60 ℃ to avoid corrosion on the uncoated plates.

Paint formulation:

A lot of researchers claimed that paint contained 60% pigment by weight give great protection towards corrosion. Therefore, the formulation of paint was started at 10, 20 and 30% PVC. Kaolin was used as an extender pigment and the composition of kaolin was fixed at 5% from PVC value. Table 1 shows the ingredients with its quantity in the formulated paint for comparison purpose. While the percentage of pigments content in formulated paints are displayed in Table 2.

Painting techniques:

After the substrate was properly cleaned and dried, it is then ready for the painting process. We employed ‘dry-on-dry’ technique, in order to have a smooth coating surface on the metallic plates. In this technique, one coat is painted and the next coat will apply after the previous coat dry completely. The thickness of coating on the substrates was varied based on the layer of coating that being applied (1, 3 and 5 layers). Painting process was conducted in the temperature ranges between 20-25ºC to achieve optimum and similar coating. While painting, the amount of paint that being used for coat need to be same as previous coat in order to have similar layer of coating. After that, the samples will be dried at ambient temperature for 1-2 hours.

Morphology observation:

Morphology of the coated substrate is studied using Scanning Electron Microscope (SEM) for analysis on the surface of coating before and after immersed in sodium chloride solution. The immersion was take place for 30 days to give a better penetration of corrosive solution.

Electrochemical polarization curve measurements:

Corrosion behaviour of coated substrate at various coating thickness was studied using electrochemical polarization curve measurements. In order to investigate the electrochemical behaviour of the coated substrate, polarization measurement was performed using electrochemical measurement system. The coated substrate sample is used as the working electrode. Platinum electrode is employed as a counter electrode, and Ag/AgCl/KCl (saturated) is used as a reference electrode. Then, all the three electrodes were connected to the test solution or electrolyte (NaCl solution). Polarization curve measurements of a sample are obtained in 3 wt./wt. % NaCl solution from -1.0V to 1.0V of potential.

Table 1: Ingredients in the formulated paint with 5% kaolin.

Sample	Layers of Coating	Pigment Volume Concentration, PVC (%)	Epoxy Resin (g)	Zinc Oxide (g)	Kaolin (g)
P1	1	10	5	2.65	0.15
P2	3	10	5	2.65	0.15
P3	5	10	5	2.65	0.15
P4	1	20	5	6.20	0.32
P5	3	20	5	6.20	0.32
P6	5	20	5	6.20	0.32
P7	1	30	5	10.50	0.55
P8	3	30	5	10.50	0.55
P9	5	30	5	10.50	0.55
S1	1	Commercialized spray			
S2	3	Commercialized spray			
S3	5	Commercialized spray			

**PVC = $(V_{pigments} / (V_{pigments} + V_{binder}))$ X 100

Table 2: Percentage of zinc and kaolin content in the formulated paint

Pigment Volume Concentration, PVC (%)	Zinc Content (w/w %)[a]	Kaolin Content (w/w %)[b]	Total Pigments Content (w/w%)[c]
30	65	3.43	68.85
20	54	2.82	56.52
10	34	1.92	35.90

**c = a + b

RESULT AND DISCUSSION

Immersion Test in Sodium Chloride Solution:

After 30 days of immersion in NaCl solution, coatings with commercial spray showed a significant rate of corrosion on the surface of the coating. The 1-layer of coating had white corrosion product and some brown rust on the surface of the coating. The white corrosion product was all over the surface of coating and sealed the cut which located on the center of the coating. Basically, the white corrosion product was from commercialized spray, while the brown rust was from production of corrosion from the steel. Commercialized spray had an active corrosion pigment and cathodic protection behaviour to block or seal the steel from corroded by using its corrosion product. As described from previous study (Kalendova, 2003), the white corrosion ($Zn(OH)_2$ or ZnO) will grow as a result of the oxidation of zinc particles. However, the existence of brown rust on the surface of coating proved that1-layer coating was too thin and insufficient to protect the steel from corroded (Figure 1 (a)). It showed that the strong diffusion of iron corrosion products from the steel to the top of coating had occurred (Shi *et al*., 2011).

The white color of corrosion product appeared on the surface of 3-layers of coating. The white color of corrosion product seems perfectly sealed on the cut part. Small amount of brown rust on the upper side of coating comes from the back side of the steel because it is not covered by the spray at the back (Figure 1 (b)). It showed that commercialized spray protects the steel from corrosion by its two different mechanisms. The first one is by sacrificial cathodic protection. The active pigment inside the spray sacrificed itself to corrode. Secondly, after the corrosion of active pigment from the spray, its white corrosion product which is the oxide layer will seal the pores on the surface of coating. This is called as barrier protection. The nature of this barier has been subject to the researchers over many years in the field of protective organic coatings.

The 5-layers of coating showed the worst. There were white and brown corrosion product from the spray and steel respectively (Figure 1 (c)). The brown rust covered the cut part. It showed that there was not enough barrier protection from the white corrosion product. As shown in Figure 1 (d), the coating was swollen and the surface of coating was broken. This proves that too much coated will make the coating available to absorb the water from the solution, thus make it penetrate through the surface of the steel and corroding the steel. The absorption of penetrating solution occurred on the porous film (Shreepathi *et al*., 2010).

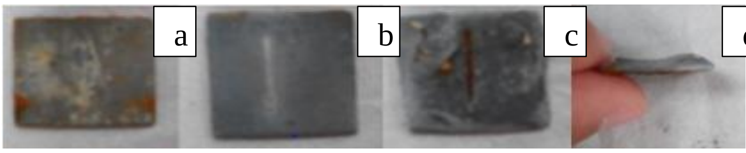

Fig. 1: The commercial spray after 30 days immersed in NaCl solution. ((a) 1 layer (S1), (b) 3 layers (S2), (c) 5 layers (infront view) (S3) and (d) 5 layers (side view) (S3)).

Figure 2 showed the brown color of steel corrosion product appeared only on the sides of the steels. This is from the corroded steel at the back of the substrate. On the surface of the coating with 30 % PVC (Figure 2 (a)), there were some changes in color of coating. While for 20 % and 10 % PVC (Figure 2 (b) and (c)), the color of the coating still remains the same. There was no swollen coating as commercialized spray. The existence of more pigments than epoxy resin in the paint gave a porous surface to the coating of 30% PVC. Since the total pigments percentage not exceed 70% by weight, therefore the porosity on the surface of the coating was not severe. The epoxy resin still capable to cover up the pores. While for 20% and 30% PVC, obviously the epoxy resin was being a protector than the pigments itself. These formulation does not need cathodic protection from the zinc because epoxy resin used its barrier protection to protect the steel from being corrode. Previous study (Shreepathi., 2010) proved that formulation with low zinc content provided good barrier protection which came from epoxy resin.

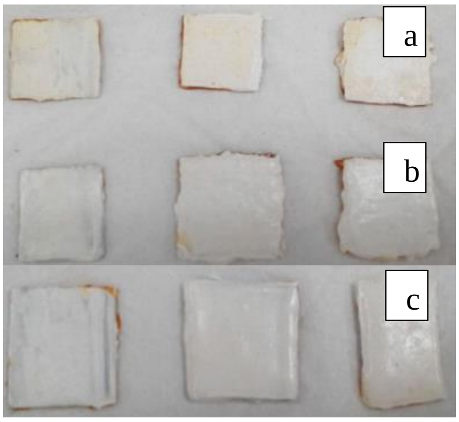

Fig. 2: The formulated paint after 30 days immersed in NaCl solution. (a: 30% PVC, b: 20% PVC and c: 10% PVC). (From left to right: 1, 3 and 5 layers of coating).

Morphological observation:

The SEM micrographs of less corroded sample before and after immersed in NaCl solution for commercialized spray and formulated paint are shown in Figure 3.

The results show that the particles of an active pigment in commercialized spray (S2) are appropriately in contact with each other. The different size of particles showed that there is an extender pigments in the spray. The presence of multiple particles size thus reducing the voids space. Figure 3 (b) showed that the corrosion products had covered and sealed the voids. It is expected that the size differences of particles in commercialized spray lead to a better packing, causing enhancement of barrier properties of the commercialized spray. While for sample P9, the particles cannot be observed clearly because of the layer of epoxy resin. The particles were covered and hold by epoxy resin. Figure 3 (d) showed that there are no corrosion products to cover the particles. Based on Figure 3, it can be seen clearly that sample S2 protects the steel with its cathodic protection and then barrier protection. Sample P9 showed that it had a barrier protection came from epoxy resin which enclosed the surface of the substrate. In this case, the zinc particles inside this formulated paint does not functioning it cathodic properties unless the NaCl solution is being absorb in the coating towards the metallic substrate, because the epoxy resin had over taken the protection by its barrier protection.

From Figure 4, the cross sectional for sample P9 showed the coating consist of microscopic flakes and hollow spheres of mullite ($3Al_2O_32SiO_2$) which from kaolin. It has excellent corrosion resistance, high thermal stability and low thermal conductivity (Morquecho *et al*., 2012). These properties proved that kaolin is a promising material as a coating. Based on Figure 4 (b), there is no corrosion product appeared in the coating. This strengthen the claim that epoxy resin provided good adhesion and corrosion resistance (Ashraf *et al*., 2015).

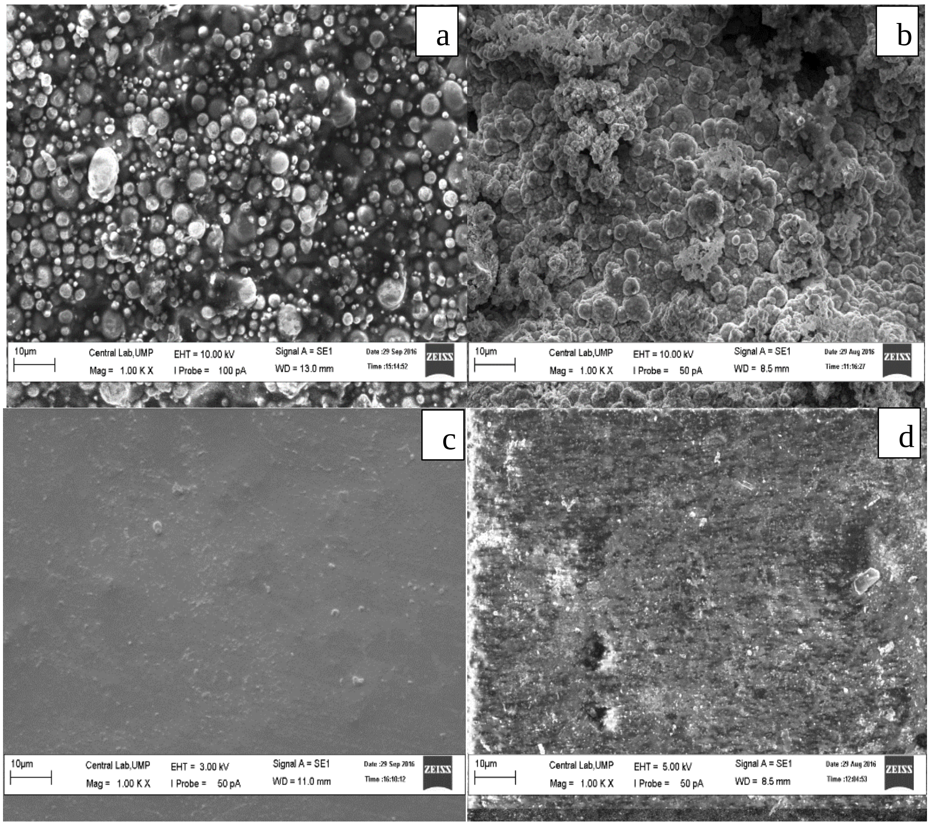

Fig. 3: SEM micrographs before and after 30 days of immersion in NaCl solution. ((a) commercialized spray (S2) before immersion test; (b) commercialized spray (S2) after immersion test; (c) formulated paint (P9) before immersion test; and (d) formulated paint (P18) after immersion test.)

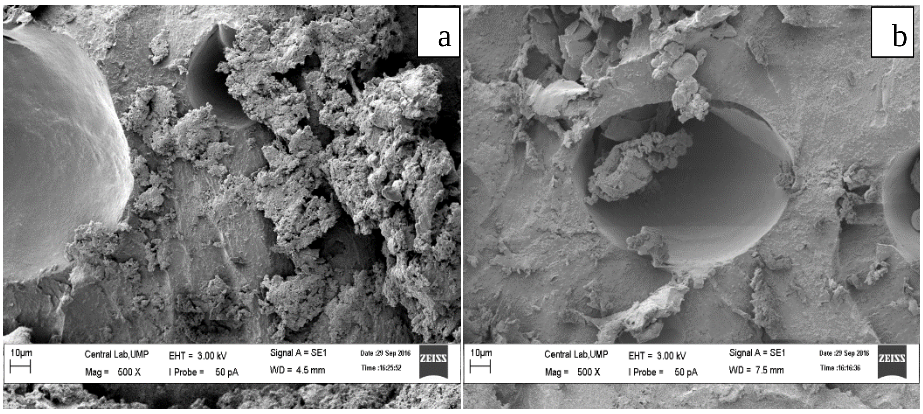

Fig. 4: SEM micrographs of sample P9 (cross sectional) before and after 30 days immersion in NaCl solution. ((a) before immersion; and (b) after immersion.)

Polarization Test in NaCl Solution:

Corrosion potential were recorded as a function of current density to evaluate the ability of cathodic protection of the coatings. The protection of steel from zinc rich paint was experimentally standardize the corrosion potential more negative than -0.78V (Shi *et al*., 2011). Above this potential, the coatings simply act as a barrier layer. Figure 6 shows the evaluation of the corrosion potentials of commercialized spray, formulated paint of 10%, 20% and 30% PVC at different layers of coating with current density.

According to Figure 5 (a), commercialized spray showed that it had cathodic protection from the active pigments. The highest E_{corr} (more negative) was -0.975V from 5 layers of coating, thus making it less noble. The active pigments in the commercialized spray actively corrode to form white corrosion products.

While for all formulated paints (Figure 5 (a), (b) and (c)), the corrosion potential was in between -0.4 to -0.5V making it less sacrificial than commercialized spray. However, the formulated paint of 10% PVC (Figure 5 (b)) the 5 layers of coating had the lowest current density approximately $1.5x10^{-8}$ A/cm^2 which lower than commercialized spray. The low value of current density basically showing that coating also can behave as a good protection from steel corrosion. This low value of current density mainly come from barrier protection of epoxy resin (Shreepathi *et al*., 2010). The lower zinc content in the formulated paints make it the lower galvanic action (Fragata *et al*.,1993). Therefore, these formulated paints behave as a barrier protection. The shape of the experimental curve for 10% PVC with 5 layers of coating indicates that the working electrode passive spontaneously when immersed in the corrodent at E_{corr} -0.5V. The current increased sharply indicates that induced anodic polarization to 1.0 V (potential) was insufficient to result in pitting (Flitt and Schweinsberg, 2005).

PVC of 30% (Figure 5 (d)) showed that the trend of polarization curve obviously differs from others. In this case, the E_{corr} was in between of -0.4 to -0.5V. The subsequent polarization curve in the more positive direction from E_{corr} in between 0 to -0.2V was from localised corrosion (Flitt and Schweinsberg, 2005). This means that the substrate undergoes spontaneous passivation in the corrodent and followed by induced pitting.

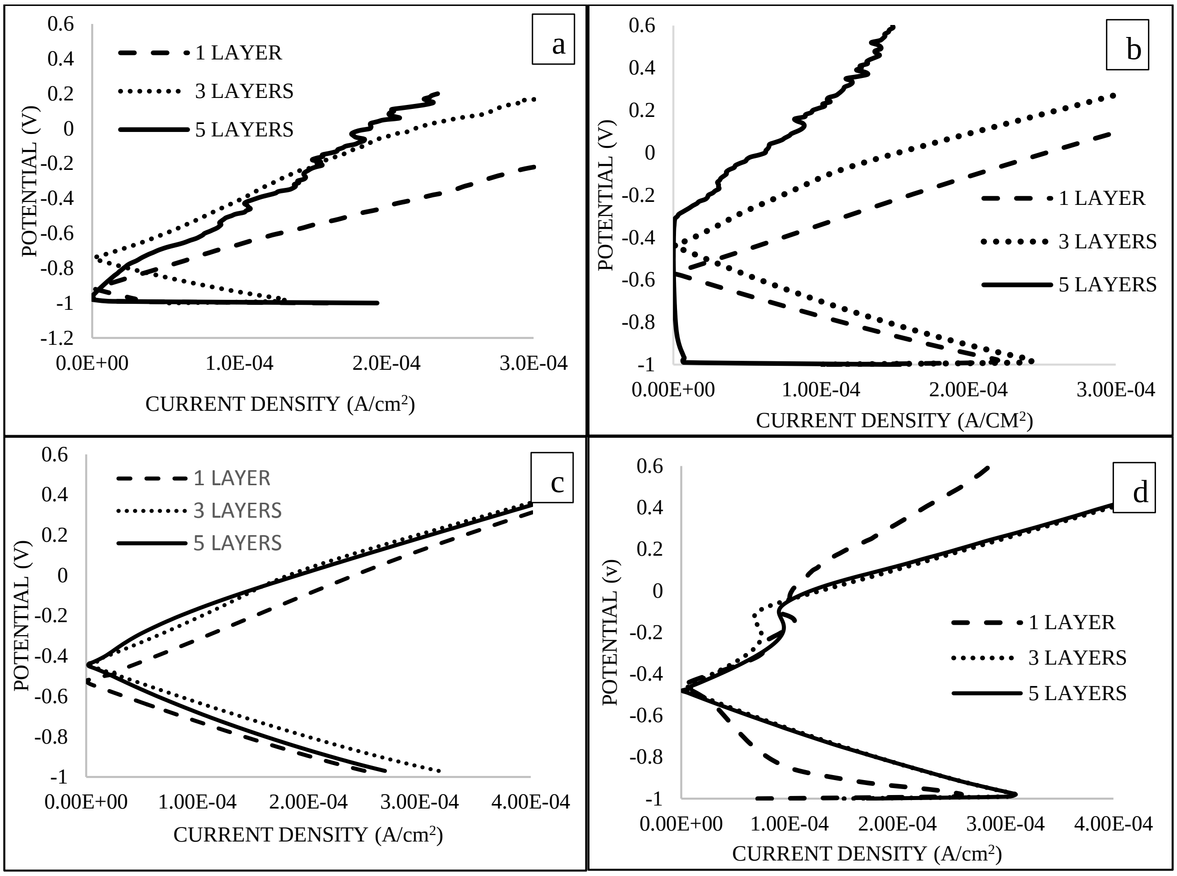

Fig. 5: Polarization measurement of commercialized spray and formulated paint. ((a) commercialized spray, (b) 10% PVC, (c) 20% PVC and (d) 30% PVC).

Table 3 showed the electrochemical properties and corrosion rates of the sample. A system's polarization resitance, R_p, was calculated by using the Stern-Geary equation (equation (1)). While equation (2) was used to calculate corrosion rate in terms of penetration rate, $\bar{\upsilon}_{p.}$

$$R_p = \frac{B}{i_{corr}} \quad (1)$$

$$\bar{\upsilon}_p = \mu_{eq} K_p \frac{i_{corr}}{\rho} \quad (2)$$

According to Table 3, P18 had the lowest current density with the lowest corrosion rate. Its corrosion rate was lower than commercialized spray even though it had less negative E_{corr} value. This show that barrier protection from the epoxy resin much better than cathodic protection from an active pigment in commercialized spray. Table 3 clearly showed that as the amount of epoxy resin increased, the corrosion rate also increased.

Table 3: Electrochemical parameters and corrosion rates

Sample	Anodic Slope, Ba	Cathodic Slope, Bc	Proportionality Constant, B	Current Density, Icorr (A/cm^2)	Linear Polarization Resistance,Rp	Corrosion Rate (mm/yr)
S1	2962.96	1666.67	463.77	5.40 X 10^{-6}	8.59 X 10^{3}	0.063
S2	2003.82	1521.74	376.05	5.00 X 10^{-6}	7.52 X 10^{3}	0.058
S3	2403.85	1875.00	457.99	5.30 X 10^{-7}	8.64 X 10^{4}	0.0062
P10	284.20	1200.00	99.90	3.80 X 10^{-6}	2.63 X 10^{3}	0.044
P11	482.85	275.86	76.33	3.25 X 10^{-6}	2.35 X 10^{3}	0.038
P12	793.65	307.69	96.40	3.20 X 10^{-6}	3.01 X 10^{3}	0.037
P13	300.17	483.33	80.51	4.50 X 10^{-6}	1.79 X 10^{3}	0.052
P14	314.25	261.90	62.11	3.20 X 10^{-6}	1.94 X 10^{3}	0.037
P15	84.32	888.89	33.48	5.50 X 10^{-6}	6.09 X 10^{3}	0.064
P16	557.77	628.14	128.45	2.70 X 10^{-6}	4.76 X 10^{3}	0.031
P17	327.46	1025.64	107.92	2.00 X 10^{-6}	5.40 X 10^{3}	0.023
P18	173913.00	606750.10	58769.30	1.50 X 10^{-8}	3.92 X 10^{8}	0.00018

Conclusion:

The corrosion behaviour of the formulated paint and commercialized spray on the metallic substrate has been studied. The 30 days immersion test in NaCl solution show that the formulated paints protect the steel by barrier protection while commercialized spray by cathodic protection and followed by barrier protection. The white corrosion product grew on the surface of the commercialized spray to seal the pores on the coating surface. Polarization measurement indicate that the corrosion potential of formulated paints less negative than commercialized paint and prove that commercialized spray less noble than formulated paints. This work suggests that the best layers of coating or thickness, should be 5-layers to avoid insufficient protection and penetration of water from the solution.

ACKNOWLEDGEMENT

The author acknowledges the financial support from the RACE grant RDU 1403141 and RDU 151308.

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